भारतीय मानक

Indian Standard

IS 8294 : 2023

एम.सी.पी.ए. का तरल अमीन लवण — विशिष्टि

(पहला पुनरीक्षण)

Liquid Amine Salts of MCPA — Specification

(First Revision)

ICS 65.100.20

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May 2023 **Price Group 5**

Pesticides Sectional Committee, FAD 01

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Pesticides Sectional Committee had been approved by the Food and Agriculture Divisional Council.

Formulations of amine salts of 4-chloro-2-methylphenoxy acetic acid (MCPA) are used extensively in the control of weeds- in cereal crops, grassland and turf.

This standard was first published in 1976. In this revision, the standard has been brought into latest style and format of Indian Standards, and references to Indian Standards wherever applicable have been updated. It also incorporates one amendment issued to this standard.

In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968, and the rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

The composition of the committee responsible for the formulation of this standard is listed in Annex E.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 2022. 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LIQUID AMINE SALTS OF MCPA — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for liquid amine salts of 4-chloro-2-methylphenoxy acetic acid (MCPA).

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below:

IS No.	*Title*
IS 460 (Part 1) : 2020	Test sieves — Specification: Part 1 Wire cloth test sieves (*fourth revision*)
IS 1070 : 1992	Reagent grade water — specification (*third revision*)
IS 8190 (Part 2) : 1988	Requirements for packing of pesticides: Part 2 Liquid pesticides (*second revision*)
IS 10627 : 1983	Methods for sampling of pesticide formulation

3 REQUIREMENTS

3.1 Description

The product shall consist of an aqueous solution containing MCPA potassium, sodium or amine salts, including mixtures, as the only active ingredients, together with any necessary formulants. The material shall be free from visible suspended matter and sediment.

3.2 The material shall also comply with the requirements given in Table 1.

3.2.1 When determined by the method prescribed in Annex A of this standard, the observed extractable acids including MCPA content percent by mass of any of the samples shall not differ by more than ± 5 percent as allowable tolerances. The actual value of the technical material in the formulation shall be calculated to the two decimal places and then rounded off to the one decimal place before applying the tolerance.

4 PACKING

4.1 The material shall be packed in clean and dry containers made of mild steel properly and suitably lacquered from inside. For packs of 10 litres or less, containers made of tin plate properly and suitably lacquered from inside may also be used. The containers shall also comply with the general requirements as stipulated in **2** of IS 8190 (Part 2).

5 MARKING

5.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name and address of the manufacturer;
c) Date of manufacture;
d) Date of expiry;
e) Batch number;
f) Net quantity;
g) Nominal MCPA content, percent (*m/m*);
h) Cautionary notice as worded in the *Insecticides Act*, 1968, and Rules framed thereunder; and
j) Any other information required under the *Legal Metrology* (*Packaged Commodities*) *Rules*, 2011.

5.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 SAMPLING

When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 within 90 days of its manufacture. When the material is offered for inspection after 90 days of its manufacture. sampling shall be done as prescribed in IS 10627. However, the criteria for conformity of the material when over the declared nominal value and given

under tested, shall be the limits of tolerances, as applicable **3.2.1** of the standard.

7 TESTS

7.1 Tests shall be carried out as prescribed in col (4) of Table 1.

7.2 Quality of Reagent

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Table 1 Requirements for Liquid Amine Salts of MCPA
(*Clause* 3.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to Annex
(1)	(2)	(3)	(4)
i)	Extractable acids including MCPA (expressed as 4-chloro-2-methylphenoxy acetic acid), percent by mass	40	A
ii)	Free phenols (expressed as 4-chloro-2-methylphenol), percent by mass, Max	1.5	B
iii)	Coarse material, water insoluble percent by mass, Max	0.1	C
iv)	Hydrogen ion concentration (*p*H)	7.0 to 9.0	D

ANNEX A
[*Table* 1, *Sl No.* (i)]

DETERMINATION OF MCPA AND OTHER EXTRACTABLE ACIDS CONTENT

A-1 PRINCIPLE

The acids are converted to their sodium salts with sodium hydroxide, acidified with hydrochloric acid, extracted with diethyl ether, and then titrated with standard sodium hydroxide using phenolphthalein.

A-2 APPARATUS

A-2.1 Separating Funnels — 250 ml capacity

A-2.2 Conical Flasks — 250 ml capacity

A-2.3 Water Bath — maintained at 80 °C to 85 °C

A-3 REAGENTS

A-3.1 Ethanol — 95 percent (*v/v*).

A-3.2 Standard Sodium Hydroxide Solution — 0.1 N

A-3.3 Sodium Hydroxide Solution — 2 N

A-3.4 Hydrochloric Acid Solution — 5 N

A-3.5 Phenolphthalein Indicator Solution — one percent (*m/v*) in 96 percent ethanol

A-3.6 Diethyl Ether

A-4 PROCEDURE

A-4.1 Weigh accurately, 0.8 g to 1.0 g of the sample and transfer, quantitatively to a separating funnel using 20 ml of sodium hydroxide solution (2 N). Shake to dissolve, and add 5 drops of phenolphthalein indicator solution, followed by sufficient hydrochloric acid to make the solution acidic. Then add 2 ml excess hydrochloric acid. Extract the solution with three successive portions of 50 ml diethyl ether and combine the extracts.

A-4.2 To check whether all the acids have been extracted carry out a fourth extraction with ml diethyl ether, and wash with not more than 10m1 of water. Transfer this extract to the conical flask. Evaporate the ether on water bath and dissolve the residue in 50 ml ethanol. Dilute the solution with 20 ml distilled water, recently boiled and cooled and titrate with standard sodium hydroxide solution using phenolphthalein.

A-4.3 If the titration of the fourth extract takes more than one drop of the standard sodium hydroxide solution, then carry out a fifth extraction, and when the conditions for complete extraction of the acids have been established, repeat the analysis. If the fourth extract does not require more than one drop of standard sodium hydroxide solution, then it may be assumed that three extractions are adequate (**A- 4.1**). Discard the aqueous layer and wash the combined extracts with three successive portions of 10 ml water. Combine these water washes and extract with 15 ml diethyl ether. Discard the aqueous layer and combine the ether layers. Transfer the ether extracts to the conical flask and proceed as in **A-4.2**.

A-5 CALCULATION

A-5.1 MCPA and other extractable acids, percent by mass = $\frac{20.06 \times N \times V}{M}$

where

N = normality of the standard sodium hydroxide solution;

V = volume, in ml, of standard sodium hydroxide solution used; and

M = mass, in g, of the sample taken for the test.

ANNEX B
[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF FREE PHENOL

B-1 PRINCIPLE

B-1.1 The absorbance of an ethanolic-ammonia solution of the sample is determined, after adding 4-aminophenazone and potassium ferricyanide solutions.

B-2 REAGENTS

B-2.1 Ammonia Solution — 0.05 N

B-2.2 Ethanol

B-2.3 Solution A — Dissolve 100 mg 4-chloro-2-methylphenol in 10 ml acetone and dilute to one litre with distilled water. One ml of this solution contains 100 µg phenol.

B-2.4 Solution B — Dissolve, 0.5 g pure MCPA, free from phenols in 50 ml of ethanol, add 90 ml of 0.05 N ammonia solution and then dilute to one litre with distilled water.

B-2.5 4-AminopheDazone Hydrochloride Solution — 0.2 percent (*m/v*), aqueous solution

B-2.6 Potassium Ferricyanide Solution — 0.4 percent, aqueous solution, freshly prepared

B-3 PROCEDURE

B-3.1 Calibration

With a micro burette, transfer 0.2 ml, 0.4 ml, 0.5 ml, 0.6 ml, 0.8 ml, 1.0 ml, and 1.2 ml of Solution A into 7 stoppered measuring cylinder and make up the volume in each to 10 ml with Solution B. Pipette 5 ml ammonia solution into each, mix the contents, add 5 ml of 4-aminophenazone hydrochloride solution, and mix again. Finally, add 5 ml potassium ferricyanide solution, shake vigorously for 1 minute, and after 5 min to 10 min measure the absorbance in a l-cm cell using distilled water in the reference cell. Determine a blank on the reagents by taking 10 ml of Solution B and treating with ammonia solution, 4-aminophenazone hydrochloride and potassium ferricyanide solutions as above. Subtract the 'blank' from the reading obtained on the phenol solution. Prepare a calibration graph plotting ml of Solution A against absorbance.

B-3.2 Determination of Free Phenols in the Sample

Weigh accurately sufficient sample to contain about 0.5 g of MCPA, Dissolve in 50 ml ethanol in the volumetric flask, and add 90 ml ammonia solution, and make up to one litre with distilled water. Pipette 10 nil of this solution into a stoppered measuring cylinder and add, in turn 5 ml ammonia, 5 ml 4-aminophenazone hydrochloride, and 5 ml potassium ferricyanide solutions, shaking after each addition Continue shaking for about 1 minute and measure the absorbance after 5 min to 10 min. Prepare a blank as given under **B-2.1** and deduct it from the value obtained with the sample. Read the number of ml of Solution A equivalent to the absorbance found.

B-4 CALCULATION

B-4.1 Free phenols (expressed as 4-chloro-2-methylphenol), percent by mass = $\frac{X}{M}$

where

X = volume, in ml, of solution A equivalent to the absorbance found; and

M = mass, in g, of the sample taken for the test.

ANNEX C

[*Table* 1, *Sl No.* (iii)]

DETERMINATION OF THE COARSE MATERIAL INSOLUBLE IN WATER

C-1 PROCEDURE

C-1.1 Weigh accurately, 20 g of the sample, mix with 100 ml of water in the stoppered measuring cylinder and shake the mixture for 10 minutes. Pour the solution through a 150 micron IS Sieve (*see* IS 460), washing out any residue from the measuring cylinder with water on to the sieve. Wash the residue on the sieve several times with distilled water and allow to drain. Brush the residue remaining on the sieve on to the glazed paper and then to the tared weighing bottle. Dry the weighing bottle and residue at 100 °C, cool and reweigh. Calculate the percent by mass of coarse material insoluble in water.

ANNEX D

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF HYDROGEN ION CONCENTRATION (*p*H)

D-l APPARATUS

D-l.1 *p*H Meter — Standardized against buffer of *p*H 7.0.

D-2 REAGENTS

D-2.1 Water — Redistilled from all acid resistance glass apparatus, and boiled to expel all carbon-dioxide just before use.

D-2.2 Phosphate Buffer Solution — 0.025 M, Dissolve 3.402 g of potassium hydrogen phosphate and 3.549 g of sodium hydrogen phosphate in water.

D-3 PROCEDURE

D-3.1 Take 25 ml of the sample in a small beaker, Determine the *p*H of this solution using the *p*H meter.

ANNEX E

(*Foreword*)

COMMITTEE COMPOSITION

Pesticides Sectional Committee, FAD 01

Organization	*Representative*(*s*)
Directorate of Plant Protection Quarantine and Storage, Faridabad	DR RAVI PRAKASH (***Chairperson***)
All India Biotech Association, New Delhi	SHRI SAURABH SINGHAL SHRI SHAH JI DHAR (*Alternate*)
Central Insecticide Board and Registration Committee, Faridabad	SECRETARY DR VANDANA SETH (*Alternate*)
Central Insecticide Laboratory, Faridabad	DR ARCHANA SINHA SHRI SUBHASH CHAUDHARY (*Alternate*)
Consumer Guidance Society of India, Mumbai	SHRI SITARAM DIXIT DR. M. S. KAMATH (*Alternate*)
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IDMA Laboratories Ltd, Chandigarh	DR INDRA RAI
Indian Agricultural Research Institute, New Delhi	DIRECTOR
Indian Institute of Packaging, Mumbai	DR TANWEER ALAM
Indian Pest Control Association, New Delhi	SHRI UDAYAN GHOSH
Institute of Pesticide Formulation Technology, Gurgaon	DR M. VAIRAMANI
Ministry of Agriculture, Department of Agriculture, Chennai	JOINT DIRECTOR OF AGRICULTURE (RES) DEPUTY DIRECTOR LAB (*Alternate*)
National Centre for Integrated Pest Management, New Delhi	DR SUMITRA ARORA

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Member Secretary
SHRI KULDEEP MITTAL
SCIENTIST 'B'/ASSISTANT DIRECTOR
(FOOD AND AGRICULTURE), BIS

Bureau of Indian Standards

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Review of Indian Standards

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